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## Assessment on potential utilization of Ariake clay as a landfill barrier material

### Evaluation sur l'utilisation potentielle de l'argile d'ariake comme materiel de barriere de remblai

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#### ABSTRACT

This paper presents an investigation on the potential utilization of Ariake clay from Kyushu of Japan as a landfill barrier material in landfills. Two heavy metals,  $Cd^{2+}$  and  $Pb^{2+}$  were selected as key contaminants. A series of batch tests were performed to investigate the effects of solid-solution contact time, solid: solution ratio and pH of solution on the sorption of two heavy metals on the Ariake clay. The batch tests results show that along the three investigated factors, the solid: solution ratio seems to be the crucial factor that controls the sorption of  $Cd^{2+}$ . A laboratory diffusion test was performed and the effective diffusion coefficients and partition coefficients of  $Cd^{2+}$  and  $Pb^{2+}$  were back-calculated using a available commercial program. The back-calculated partition coefficient of  $Cd^{2+}$  from advection-diffusion test was found to have a value close to the one derived from the batch test at the soil: solution ratio of 1: 1.3. This result again announces the importance of selecting a proper solid: solution ratio to performing a batch test to determine sorption parameters. The diffusion test shows that the  $Cd^{2+}$  and  $Pb^{2+}$  were highly retained on the Ariake clay specimen indicating that Ariake clay may be used as a good barrier material to attenuate heavy metals in landfills.

#### RÉSUMÉ

Ce document présente une recherche sur l'emploi possible de l'argile d'Ariake du Kyushu (Japon) comme matériau de rétention dans les décharges. Deux métaux lourds,  $Cd^{2+}$  et  $Pb^{2+}$ , ont été sélectionnés comme contaminants principaux. Une série de tests groupés ont été conduits pour examiner les effets du temps de contact solution-solide, le ratio de solution-solide et le pH de solution sur l'absorption des deux métaux lourds par l'argile d'Ariake. Les résultats de ces tests montrent que parmi les trois facteurs étudiés, le ratio solution-solide semble être le facteur prédominant contrôlant l'absorption du  $Cd^{2+}$ . Un test de diffusion a été effectué en laboratoire et les coefficients de diffusion effective ainsi que les coefficients de répartition de  $Cd^{2+}$  et  $Pb^{2+}$  ont été calculés par analyse inverse à l'aide d'un logiciel du commerce. Le coefficient de  $Cd^{2+}$ , calculé par analyse inverse depuis les tests d'advection-diffusion, s'est révélé d'une valeur proche de celle dérivée des tests groupés sur le ratio solution-sol, de rapport 1:1.3. Ce résultat montre l'importance d'un ratio de solution-solide adéquat pour réaliser un test groupé visant à déterminer les paramètres d'absorption. Le test de diffusion montre que le  $Cd^{2+}$  et le  $Pb^{2+}$  ont été fortement retenus par le spécimen d'argile d'Ariake, ce qui indique que celle-ci peut être utilisée comme un excellent matériau de rétention des métaux lourds dans les décharges.

#### 1 INTRODUCTION

Heavy metals such as  $Cd^{2+}$  and  $Pb^{2+}$  are commonly found in leachates from landfills. Due to the toxicity and migration, these heavy metals may cause impact on the underlying aquifer and therefore pose potential risk to human health and surrounding environment. One way for reducing the risk is to use soil barriers to limit and control heavy metals migrated from landfills. To evaluate application of clays for barrier materials, some parameters, such as sorption and diffusion coefficients of heavy metals, need to be clarified. A simple and easy way to investigate sorption behaviors of clays is to use batch test (ASTM, 1993; Shackelford and Daniel, 1991). Factors controlling sorption of heavy metals on clays, such as soil-solution contact time and solid: solution ratio (hereinafter called  $s/w$  (g/mL)), should be investigated (Lecite et al., 2003). To determine diffusion coefficients of certain heavy metal species in clays, usually column test, either diffusion tests or diffusion-advection tests, are required (Rowe et al., 1988; Shackelford and Daniel, 1991).

There is a tendency in Japan to build waste sites at coastal areas (Kamon and Katsumi, 2001), and regional marine clays of interest may provide source for being potentially used as soil barrier materials. Du et al. (2000) indicated that the Ariake clay, which is a typical regional marine clay located in the Kyushu of Japan, seemed to be a good barrier material because of its high sorption capacity. However, so far rather little has been published concerning assessment on potential use of Ariake clay as a barrier material to reduce contamination risk of heavy

metals. This results in much uncertainty in using Ariake clay as barrier in industrial landfills where heavy metals are commonly encountered.

The main purpose of this study is to evaluate the potential application of Ariake clay to a barrier material in landfills. For this purpose, following folds have been presented: 1) batch tests showing the effects of solid-solution contact time,  $s/w$  and pH of solution on the sorption of two heavy metals,  $Cd^{2+}$  and  $Pb^{2+}$ ; 2) laboratory diffusion test for determining the diffusion coefficients and partition coefficients of  $Cd^{2+}$  and  $Pb^{2+}$  in the Ariake clay. The back-calculated partition coefficient of  $Cd^{2+}$  is compared with the one derived from the batch tests. The importance of selecting proper  $s/w$  to performing batch tests to determine sorption parameters is discussed.

#### 2 MATERIALS DESCRIPTION

In this study, the Ariake clay was sampled at a 3 m depth from the ground surface at the Kawasoe Machi, Saga Prefecture, Kyushu of Japan. Some geotechnical and chemical properties are shown in Table 1.

The synthetic leachate used in this study was selected as a solution containing  $CdCl_2$ ,  $PbCl_2$  and KCl. To investigate the influence of solid-solution contact time, the initial concentrations of  $Cd^{2+}$  and  $Pb^{2+}$  were 58.1 mg/L and 30.2 mg/L respectively, and the initial pH of solution was about 6. To investigate the effect of  $s/w$  on sorption, the initial concentrations of  $Cd^{2+}$

Specific gravity, $G_s$	2.68
Natural water content, $w_n$ (%)	173
Liquid limit, $w_L$ (%)	115
pH	7.87
Clay particle fraction ( $<2\mu\text{m}$ ) (%)	46
Silt particle fraction (%)	49
Sand particle fraction (%)	5
Primary clay mineral	smectite
CEC (meq/100 g)	34
Specific surface area ( $\text{m}^2/\text{g}$ )	69
Ignition loss (%)	10.2

and  $\text{Pb}^{2+}$  for each of the four considered cases presented later in this study were (10.7, 25.1, 40.1 and 69.6 mg/L) and (1.4, 16.2, 19.7 and 27.1 mg/L) respectively. The initial pH of solution varied in the range of 5.64 to 6.08.

### 3 BATCH TEST

#### 3.1 Test method

The batch test was performed according to the standard testing methods prescribed by ASTM (1993) and US EPA (1987). Generally, it consists of mixing the soil and synthetic leachate in a closed container at specific  $s/w$  for specific time. Then a sample of the soil-solution slurry from the container was centrifuged for 30 minutes at 3000 rpm. After that, the concentrations of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  in the supernatant sampled from the tube were measured using a HITACHI Z-6000 atomic adsorption spectrophotometer, and termed equilibrium concentrations,  $C_e$ . To investigate the influence of soil-solution contact time,  $s/w$  was set as 1: 20 and contact time was 1h, 2h, 4h, 6h, 9h, 24 h and 48h. To investigate the effect of  $s/w$ , four cases were considered by setting  $s/w$  at 1: 20, 1: 10, 1: 4 and 1: 1.3 with contact time of 24 h. All of the tests noted above were performed at a controlled constant laboratory temperature of 22°C.

The chemical analysis of the batch-type tests was plotted in the form of the adsorption isotherm or equilibrium concentration,  $C_e$  (mg/L), versus sorbed concentration,  $q$  ( $\mu\text{g}/\text{g}$ ). The sorbed concentration,  $q$ , was determined according to the following equation:

$$q = \frac{(C_0 - C_e)V_{sol}}{M_s} \times 1000 \quad (1)$$

where  $C_0$  = the initial concentration of  $\text{Cd}^{2+}$  or  $\text{Pb}^{2+}$  (mg/L),  $V_{sol}$  = the volume of the solution (L) and  $M_s$  = the soil mass (g, oven-dried basis).

To predict the relationship between sorbed and equilibrium concentrations in batch tests, the Freundlich isotherm equation was adopted as expressed by Eq (2):

$$q = K_f C_e^N \quad (2)$$

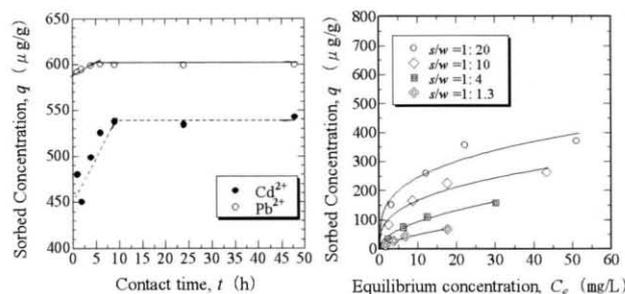


Figure 1. Effect of contact time on sorption of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$ .

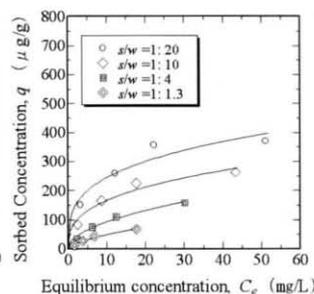


Figure 2. Effect of  $s/w$  on sorption of  $\text{Cd}^{2+}$  on the Ariake clay.

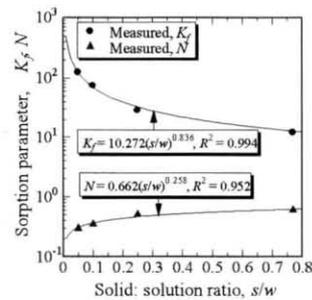


Figure 3. Change of  $K_f$  and  $N$  with  $s/w$  for  $\text{Cd}^{2+}$

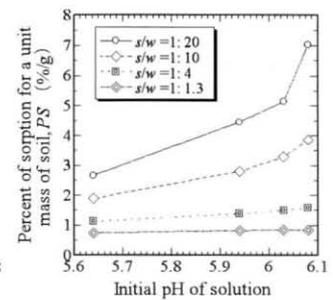


Figure 4. Change of  $PS$  of  $\text{Cd}^{2+}$  with initial pH of solution.

in which  $K_f$  and  $N$  are the Freundlich sorption parameters adjusted to fit Eq. (2) to the experimental data.

The percent of sorption for a unit mass of soil,  $PS$  (%/g), is defined as:

$$PS = \frac{(1 - C_e/C_0)}{M_s} \times 100 \quad (3)$$

#### 3.2 Test results and discussion

Figure 1 shows the change of sorbed concentrations versus contact time for  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$ . It can be seen that it took about 9h and 6h for  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  respectively to reach the equilibrium conditions. When the sorption kinetics is concerned for modeling the transport of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  in Ariake clay barrier in landfills, 24 h is enough to achieve equilibrium. From Fig. 1, it is also clear that Ariake clay sorbed larger amount of  $\text{Pb}^{2+}$  relative to  $\text{Cd}^{2+}$  indicating that the selectivity of  $\text{Pb}^{2+}$  was higher than that of  $\text{Cd}^{2+}$ .

The adsorption isotherms for  $\text{Cd}^{2+}$  at four different  $s/w$  values are depicted in Fig. 2. From Fig. 2, it is clear that sorption of  $\text{Cd}^{2+}$  largely depended on the  $s/w$ . With increasing  $s/w$ , the amount of sorbed  $\text{Cd}^{2+}$  decreased. The largest amount of sorbed  $\text{Cd}^{2+}$  was observed at  $s/w = 1: 20$  while lowest at 1: 1.3. The derived Freundlich sorption parameters,  $K_f$  and  $N$  are plotted versus  $s/w$  in Fig. 3. It is clear that at low value of  $s/w$ ,  $<1: 4$ ,  $K_f$  and  $N$  changed significantly, whereas at high value,  $>1: 4$ ,  $K_f$  and  $N$  changed slightly. The observed  $s/w$ -dependent sorption behavior indicates that with respect to the determination of the sorption parameters from the batch tests to be used for modeling transport of heavy metals in Ariake clay barrier, the effect of  $s/w$  must be considered.

For  $\text{Pb}^{2+}$ , the equilibrium concentrations in the batch tests at different  $s/w$  were almost zero, and thereby construction of the adsorption isotherms was failed. As a result, the Freundlich sorption parameters for  $\text{Pb}^{2+}$  were not able to be determined. From Eq. (1), it can be seen that the less the value of  $C_e$ , the higher the value of  $q$  will be. Therefore, this observation indirectly indicates that  $\text{Pb}^{2+}$  has higher selectivity than  $\text{Cd}^{2+}$ .

To understand the influence of pH, the relationship between the values of  $PS$  and initial pH is depicted in Fig. 4. Generally, it can be seen that with increasing pH, the value of  $PS$  increased. For the case of relative low  $s/w$  (i.e., 1: 20 and 1: 10),  $PS$  increased significantly with increasing pH whereas for the case of relative high  $s/w$  (i.e., 1: 4 and 1: 1.3),  $PS$  increased only marginally with increasing pH. This result indicates that the effect of pH on sorption may largely depend on  $s/w$ . A report of the influence of pH on sorption should be reported along with the presence of value of  $s/w$ , at least for the soil, solutions, and pH range presented in this study.

### 4 DIFFUSION TEST

#### 4.1 Test procedure

Prior to the diffusion test, the Airake clay specimen was prepared by consolidation. About 1.7 kg disturbed Airake clay with natural water content was poured into an acrylic cylinder with inner diameter of 10 cm (Fig. 5). A porous plate with thickness of 0.4 cm and porosity of 0.42 was placed above the soil. A perforated stainless steel plate with thickness of 0.4 cm and open space coverage (open space volume/total volume) of 80% was placed above the porous plate, and was connected with a stainless steel rod used to transfer the vertical pressure, 57 kPa, applied from a device similar to the odometer test apparatus. To prevent drying of the clay surface and to keep pore fluid close to the original one, about 10 mL pore water extracted from the other samples of the same clay were added on the clay periodically. During the consolidation, the valve of the plastic tubing at the base was kept open to drain the pore water. The consolidation lasted for about 1 month till the observed change in the height of specimen was marginal. The valve was then closed and the remained pore fluid above the perforated steel plate was removed immediately. The prepared synthetic leachate, a solution containing CdCl<sub>2</sub>, PbCl<sub>2</sub> and KCl, was introduced into the cylinder and the diffusion test was started. The properties of the soil specimen and solution used for the diffusion test are listed in Table 2. These parameters would be used for the diffusion test result analysis as described. Throughout the test, concentrations of Cd<sup>2+</sup> and Pb<sup>2+</sup> in the reservoir were periodically monitored by withdrawing 1 mL samples from the solution through the sampling port at three-day intervals (see Fig. 5). The same volume of distilled water as that of the sampled solution was added to the source reservoir. At the completion of the diffusion that lasted for 54 days, the solution in the cylinder was drained away and the apparatus was disassembled. The soil specimen was extruded and sliced into seven sublayers using a stainless steel thread. The pore water in each sublayer was obtained by squeezing the soil at a pressure of about 12.8 kPa for approximately 12h. The concentrations of Cd<sup>2+</sup> and Pb<sup>2+</sup> in the squeezed pore water were analyzed using a HITACHI Z-6000 atomic adsorption spectrophotometer.

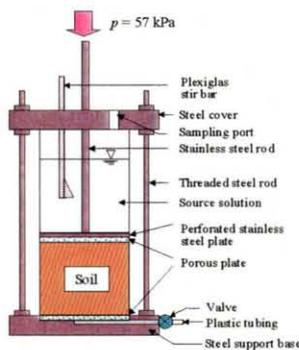


Figure 5. A schematic diagram of diffusion test apparatus.

Table 2: Properties of soil specimen and solution.

Thickness (cm)	10.2
Dry density, $\rho_d$ (g/cm <sup>3</sup> )	0.77
Porosity, $n$	0.71
Water content (%)	91
Saturation degree (%)	100
Hydraulic conductivity, $k \times 10^{-9}$ (m/s)	1.7
Height of solution, $H_f$ (cm)	10.2
Initial concentration, $C_0$ (mg/L)	61.8 (Cd <sup>2+</sup> ), 22.1 (Pb <sup>2+</sup> )
pH of solution before diffusion test	5.88
pH of solution after diffusion test	7.83
Solution collected, $q_c \times 10^{-10}$ (per area per unit time, m/s)	3.5
Duration of test (days)	54

#### 4.2 Results analysis and discussion

The migration of Cd<sup>2+</sup> and Pb<sup>2+</sup> was evaluated considering the one-dimensional governing equation for diffusive transport in saturated soil as follows:

$$(n + \rho_d K_p) \frac{\partial C}{\partial t} = n D_e \frac{\partial^2 C}{\partial x^2} \quad (4)$$

in which  $\rho_d$  is the dry density of the soil,  $n$  is the porosity of the soil,  $C$  is the concentration of contaminant at the point and time of interest,  $K_p$  is the partition coefficient and  $D_e$  is the diffusion coefficient.

The upper boundary imposed by the source solution was the finite mass condition as follows:

$$C(t) = C_0 - \frac{1}{H_f} \int_0^t f_T(\tau) d\tau - \frac{q_c}{H_f} \int_0^t C(\tau) d\tau \quad (5)$$

The base was impermeable such that the lower boundary was modelled as a zero-flux boundary condition, or:

$$f_B(t) = -n D_e \left( \frac{\partial C(t)}{\partial x} \right)_B = 0 \quad (6)$$

where  $C_0$  is the initial concentration of Cd<sup>2+</sup> or Pb<sup>2+</sup>,  $H_f$  is the height of source solution.  $q_c$  is the volume of fluid per unit cross-sectional area of the soil per unit time removed from the source solution and replaced by distilled water,  $f_T$  is the mass flux from the source into the soil as given by

$$f_T = -n D_e \frac{\partial C}{\partial x} \quad (7)$$

and  $f_b(t)$  is the mass flux entering the base at specific time  $t$ . A solution to Eqs. (3)-(7) has been given by Rowe and Booker (1985) and completed in a commercial program, POLLUTE V 6.3 as described by Rowe & Booker (1994). Using various combinations of  $D_e$  and partition coefficient,  $K_p$ , theoretically calculated curve for concentration variation with time in the source solution were generated and then compared with the experimental data. The values of  $D_e$  and  $K_p$  which together give the best fit "by eye" to the experimental data, were selected as the back-calculated values. Rowe and Booker (1985) have proved that only a single combination of  $D_e$  and  $K_p$  would give the best fit.

All of the input parameters required for POLLUTE V 6.3 are summarized in Table 2. The experimentally measured data and best-fit curves for concentration variation with time in the source solution are shown in Fig. 6. The back-calculated values of  $D_e$  and  $K_p$  for Cd<sup>2+</sup> and Pb<sup>2+</sup> are  $4 \times 10^{-10}$  m<sup>2</sup>/s and 2 mL/g, and  $4 \times 10^{-10}$  m<sup>2</sup>/s and 100 mL/g, respectively. The values of diffusion coefficients are well in the range of literature work for Cd<sup>2+</sup> of  $3.0 \times 10^{-10}$  m<sup>2</sup>/s to  $4.2 \times 10^{-10}$  m<sup>2</sup>/s (Shackelford and Daniel, 1991), indicating the test method used in this study is reasonable. From Fig. 6, it can be seen that the predicted curve for Pb<sup>2+</sup> better fit the experimental data relative to that for Cd<sup>2+</sup>. For Cd<sup>2+</sup>, after about first 31 days, a substantial drop of the concentration was observed till the end of the diffusion test. However, only the data before the elapsed time of 31 days were used for back-calculating the  $D_e$  and  $K_p$  for Cd<sup>2+</sup>. This considerable drop of concentration of Cd<sup>2+</sup> may be partly due to the increase of pH in the source solution during the diffusion test. Before starting the diffusion test, the measured pH of the solution was 5.88 whereas it increased to 7.83 at the end of the test. It may be inferred that during the test (probably after 31 days), the pH of the solution may have increased to values (e.g. pH >7) that are fa-

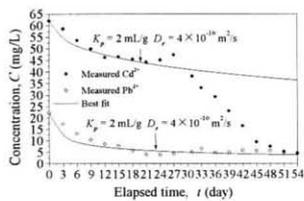


Figure 6. Concentrations of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  versus time in the source solution.

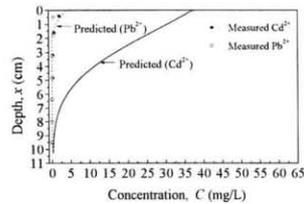


Figure 7. Concentrations of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  versus depth in the soil.

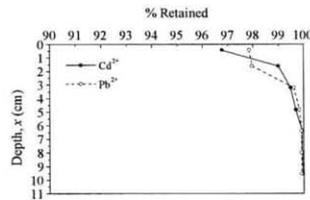


Figure 8. Retained percents of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  versus depth in the soil.

vorite for the formation of hydroxy species of Cd (e.g.  $\text{Cd}(\text{OH})_2$ ), and thereby the concentrations of  $\text{Cd}^{2+}$  decreased considerably. The modeling process used in this study does not take into account such a geochemical change and therefore the experimental concentrations were lower than the predicted concentrations after from 31 days of testing. Yong et al. (1992) indicate that that hydroxy species for Cd only began to form at  $\text{pH} \geq 7$  and at  $\text{pH} \geq 4$  for Pb. This difference may explain why a substantial drop in concentration for  $\text{Cd}^{2+}$  was observed in the source solution whereas no such drop was observed for  $\text{Pb}^{2+}$ . Due to the formation of hydroxy species for Cd, most of  $\text{Cd}^{2+}$  ions have been retained on the Ariake clay, and thereby the measured concentration distribution profile in the specimen did not match the predicted values (Fig. 7). The  $s/w$  adopted for the advection-diffusion test was about 1: 1.3. However, at the same  $s/w$  in the batch test, this increase in sorption was not observed. One of the possible reasons may be that all of the  $\text{pH}$  at equilibrium for the batch test was less than 7, and large formation of hydroxy species for Cd may not be mobilized. The retained percents of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  at different depth of specimen were calculated using Eq (8):

$$\% \text{ Retained} = (1 - C/C_0) \times 100 \quad (8)$$

The retained percents of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  distributed in the specimen are depicted in Fig. 8. It can be seen that both  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  have been greatly retained in the soil, with retained percents being larger than 95%. At depths deeper than 3 cm, the retained percents even exceeded 99%. Thereby it is concluded that Ariake clay has much high ability for retaining  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$ , indicating that it may be used as a good barrier material for attenuate heavy metals in landfills.

The back-calculated  $K_p$  is compared with that derived from the batch tests using the following equation (Shackelford and Daniel, 1991):

$$K_p = \frac{\Delta q}{\Delta C} \Big|_{C_0} = \frac{q|_{C_0} - q|_{C=0}}{C_0 - 0} = \frac{K_f C_0^N}{C_0} = K_f C_0^{N-1} \quad (9)$$

in which  $K_f$  and  $N$  are Freundlich sorption parameters. Table 3 shows the values of  $K_p$  for  $\text{Cd}^{2+}$  at different values of  $s/w$ . It can be seen that the back-calculated  $K_p$  was generally lower than those derived from the batch tests, but close to the one calculated at  $s/w = 1: 1.3$ . Here again the importance of selecting appropriate  $s/w$  for a batch test to determining appropriate sorption parameters for predicting the transport of heavy metal in clay barrier, is announced.

Table 3: Values of calculated  $K_p$  of  $\text{Cd}^{2+}$  between batch test and diffusion test

Test method	Solid: Solution	$K_p$ (mL/g)	Calculation method
Batch test	1: 20	6.8	Using Eq (9)
	1: 10	5.1	
	1: 4	3.8	
Diffusion test	1: 1.3	2.4	Back analysis
	About 1: 1.3	2.0	

## 5 CONCLUSIONS

This study presents an assessment on potential utilization of Ariake clay as a soil barrier material in landfills. The following conclusions are drawn:

- 1) The sorption of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  on the Ariake clay reached a satisfied equilibrium condition at hours of 6 and 9 respectively. This indicates that when the sorption kinetics is concerned in modeling transport of heavy metals in Ariake clay, 24 h is enough to reach equilibrium.
- 2) The amount of sorbed  $\text{Cd}^{2+}$  and effect of  $\text{pH}$  of source solution on sorption of  $\text{Cd}^{2+}$  on the Ariake clay were crucially affected by  $s/w$ , indicating that when performing batch test to derive sorption parameters, the influence of  $s/w$  must be considered.
- 3) The effective diffusion coefficients of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  determined from the diffusion test for Ariake clay are consistent well with the literature study, indicating that the testing method presented in this study is acceptable. The highly retained percents of  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  on the Ariake clay indicates that Ariake clay may be used as a good barrier material to attenuate contamination of heavy metals in landfills.
- 4) The back-calculated partition coefficient of  $\text{Cd}^{2+}$  from diffusion test was found to have a value close to the one derived from the batch test at the condition of  $s/w = 1: 1.3$ . This result again announces the importance of selecting a proper  $s/w$  to perform a batch test to derive sorption parameters.

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